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Etch Pit Studies of II-VI-Wide Bandgap Semiconductor Materials ZnSe, ZnCdSe, and ZnCdMgSe Grown on InP

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Abstract

Etch pit density (EPD) determination studies have been conducted on II-VI semiconductor materials ZnSe, ZnCdSe, and ZnCdMgSe grown on InP surfaces for the first time by using various etching solutions under different conditions of concentration, temperature, and time. We observed that for the binary, ternary, and quaternary samples, bromine-methanol solution was the only etchant with a reasonable etching effect. We could only perform an in-depth etching study for ZnSe grown on InP by using a Br:MeOH solution. Other etchants did not have an effect on the surface or morphology of the materials, and hence, a meaningful or consistent EPD could not be determined for ZnCdSe and ZnCdMgSe.

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1. Introduction

Wide bandgap II-VI compounds have applications as blue-green laser diodes [1]. Such laser structures are always grown on GaAs substrates. However, because large lattice mismatch exists, the quantum well $\text{Zn}_{1-x}\text{Cd}_x\text{Se}$ has to be thin to be pseudomorphic. Even when the compounds are pseudomorphic, the large lattice mismatch with the substrate GaAs (1.6% at $x = 0.2$) [2] produces strain in the quantum well and may cause the diodes to degrade during operation. At $x = 0.525$, the lattice constant of $\text{Zn}_{1-x}\text{Cd}_x\text{Se}$ is equal to that of InP (5.87 Å). Similarly, other ternary and quaternary alloys also can be grown lattice-matched to InP. By proper selections of materials and experimental conditions, one can grow strain-free, lattice-matched heterostructures useful for optoelectronic device applications. However, defects such as dislocations, which are produced during growth of these materials, propagate from the substrate to the nearest layer, thus reducing the performance of such devices. Therefore, the study of defect density in these materials is very important so that high-quality materials can be grown. Dislocations result from thermal stresses and mechanical deformations. For a better understanding of these deformations and defects, one can use various experimental techniques.

The chemical etching technique is very useful to determine dislocation densities and has been used by many workers [3,4] in the II-VI wide bandgap materials. It is a powerful but simple and very effective technique. Materials are sensitive to etchants under different conditions, thus helping the investigation of crystal structure, defects, and inhomogeneities. Chemical etching distinguishes different regions of the crystal for quality. It is also widely used for cleaning the surface of a semiconductor for processing applications. Defects may occur as a result of etching, so it is important to understand the consequences to the near surface region and the perturbations in the physical properties of the sample materials. In general, crystal dislocations are preferentially attacked during etching because dislocations have different chemical potential than the bulk crystal. However, very few studies of chemical etching and its effects on epilayers grown on InP have been performed. Reduced photoluminescence (PL) intensities have been reported after photochemical etching of ZnSe [5], but there is no report of any similar work done on ZnSe, ZnCdSe, and ZnCdMgSe grown on InP because of surface morphology and the difficulty in etching them, especially in the case of ternary and quaternary compounds.

2. Experiments

We grew the epitaxial layers by molecular beam epitaxy (MBE) in a Riber 2300P growth chamber. Before the growth, substrates were etched in $\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2:\text{H}_2\text{O}$ (4:1:1) for 1 to 2 min. As previously performed by Dai et al [6], we performed oxide desorption of the InP substrate by heating the substrate with an As flux impinging on the InP surface. The best results were obtained by heating the substrate quickly to $\sim 500^\circ\text{C}$, then lowering the temperature to the initial growth temperature of 170°C . Once the growth was initiated, after 1 min, the growth temperature was raised to the optimum temperature of 270°C . Under these conditions, we routinely obtained featureless, defect-free surfaces and two-dimensional nucleation of the II-VI layer on the InP substrate (as indicated by the presence of a streaky pattern of reflection high-energy electron diffraction (RHEED) throughout the nucleation and growth). The epilayer thickness was about 2 or 3 μm for most of the samples. We analyzed grown samples by low-temperature PL at 10 K using a 325-nm line of a He-Cd laser. Crystal quality was determined with x-ray rocking curve measurements. Results indicated excellent PL quality and double-crystal rocking curves.

We cut and inspected samples grown by the MBE for orientation (100) using Laue x-ray diffraction. ZnCdSe and ZnCdMgSe samples with narrow PL peaks and low full width at half maximum (FWHM) for the x-ray rocking curve measurements were chosen for etch pit density (EPD) studies. To understand surface morphology and lattice mismatch effects with varying Cd contents, we studied $\text{Zn}_{1-x}\text{Cd}_x\text{Se}$ for as-grown and etched samples. We also conducted similar studies for the quaternary $\text{Zn}_x\text{Cd}_y\text{Mg}_{1-x-y}\text{Se}$. For most of the etching experiments, indium, which was used at the back of the substrate as a glue to the substrate holder, was removed. Before the etching, samples were degreased in organic solvents without drying between steps, as explained in the following paragraphs.

Samples were soaked in trichloroethylene with ultrasonic agitation followed by rinsing in acetone and methanol. For maintaining control of the rate of etching, we prepared various concentrations of the bromine-methanol solution. Bromine solution was varied in the range of 0.05% to 0.5% by volume percent. These solutions were kept at $2 \pm 1^\circ\text{C}$ to control the reaction.

Once the setup was ready, we immersed samples in a particular concentration of Br:MeOH solution for varying times. Samples were taken out of the etching solution after the prescribed time, rinsed with deionized water, and dried by blowing with dry nitrogen gas. We used a Dektak IIA surface profile system to measure the etch depths.

In another experiment, we treated the samples in the Hahnert-Schenk [7] solution with a basic composition of 1 vol. part concentrated HF (hydrofluoric), 1 vol. part chromic acid (30 wt % K_2CrO_3 in H_2O), and 1 vol. part HCl and diluted the samples with H_2O as required. Samples were also treated for varying times with Nakagawa etch [8], which consisted of

HF:H₂O₂:H₂O, 3:2:2 v/v. In a third experiment, samples were etched in boiling NaOH (30%) [9,10] in HCl for varying times.

In another experiment, we treated samples in Polisar [11] etch for 3 min or longer. Treated samples were observed under a Nomarsky microscope, and Polaroid pictures were taken for record. Some of the samples used for the scanning electron microscopy (SEM) study, especially ZnCdSe, were treated with the Hahnert-Schenk solution, boiling NaOH, and HCl.

3. Results and Discussion

In the study conducted in this report, all the epilayers were (100) oriented. Ternary and quaternary samples were etched differently compared to the binary material ZnSe. Etching behavior of ZnSe was controllable with bromine-methanol solutions for low concentrations. As indicated earlier, ZnSe grown on InP was treated with different etchants, and we later observed the effects of the etchants under a microscope to evaluate the surface morphological changes. Table 1 gives the description of all the chemical etchants used for the described studies in the case of ZnSe grown on InP.

For ZnSe, the study using bromine-methanol etchant under varying conditions of time, concentration, and temperature proved to be very productive. As indicated in figure 1, time of immersion and changes in the bromine-methanol concentration varied the etching depth.

When the bromine-methanol concentration was changed from 0.05% to 0.25%, the etching depth changed drastically. For the 0.05% concentration, the etching was slow and almost linear. This may be due to the bromine still being active in the solution, the epilayer being available for etching by the bromine, and the temperature of the reaction being the required range to proceed. For the 0.10% and 0.25% bromine solution, the reaction was extremely active and the etching was steep. However, after we exposed the samples for 300 s in the solution, the reaction deviated. This may be due to the slowing down of active species in the reagents, the reagents closely reaching the epi depth, or the temperature of the reagents having changed due to the ongoing reaction. In general, the etching depth was proportional to the etching time. However, we observed a leveling

Table 1. Surface morphology for ZnSe grown on InP (100) after chemical etching under varying conditions of time, concentration, and temperature.

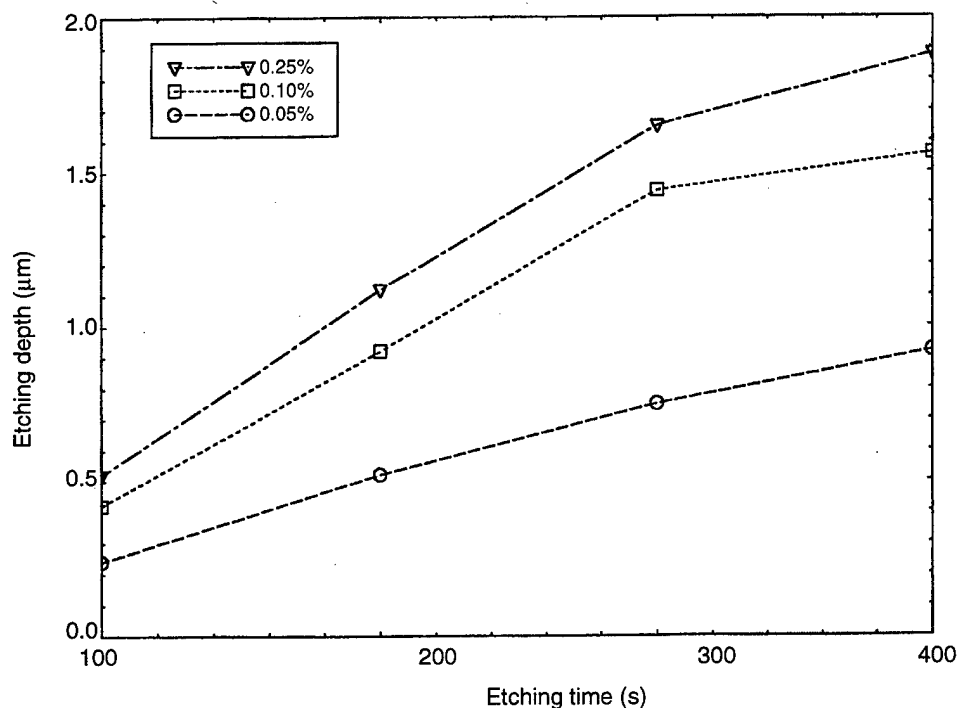
Etchant	Chemical etch	Action of etchants
Br:MeOH	0.05%, 0.10%, and 0.25%	Good etchant. Produces controllable pits. Reaction is linear up to certain etching time. EPD can be calculated.
Hahnert-Schenk solution ^a	1:1:1 HF:chromic acid:HCl	Rough surface. Few pits formed. No control.
Nakagawa solution ^b	3:2:2 HF:H ₂ O ₂ :H ₂ O	Pitted.
Boiling NaOH (30 wt %)	—	High density of pits. No control.
HCl	—	Rough surface.
Polisar (I) solution ^c	1:1:1 HNO ₃ :HCl: H ₂ O and 0.9 mL Br ₂	Rough surface.

^aI. Hahnert and M. Schenk, *J. Cryst. Growth* **101** (1990), 251.

^bK. Nakagawa, K. Maeda, and S. Takeuchi, *Appl. Phys. Lett.* **34** (1979), 574.

^cE. L. Polisar, E. M. Boinikh, G. V. Indenbaum, A. V. Vanyukov, and V. P. Schastlivii, *Izv. Vyssh. Uchebn. Zaved. Fiz.* **6** (1968), 81.

Figure 1. Etching depth against time for ZnSe grown on InP. Legend indicates various concentrations of bromine-methanol solutions.



off of the etch rate after 300 s of etching for a particular concentration, indicating the nonlinearity of the etching when etching concentration was changed. The etching depth did not double with the doubling of the concentration of the etching solution. Figure 2 shows the etching depth rate for ZnSe for different etchant concentrations of the bromine-methanol solution. The legend indicates total etching time for different concentrations of the solution.

Nonlinearity is not part of the etching process and can be attributed to other reactions that may be happening after certain concentrations. It also can be attributed to the reaction temperature rising to the point that it retards the reaction. In essence, the efficiency of etching goes down as time passes and beyond a certain concentration of the etchant solution.

For ZnSe, we observed major changes using Br:MeOH etchant under varying conditions. Reactions were meaningful and results could be obtained. Other etchants, including Hahnert-Schenk and Nakagawa solutions, did not make any drastic morphological changes to ZnSe. There were few pits formed in both cases with no control. Boiling NaOH created high density of pits, and HCl produced rough surfaces without any pattern. A Polisar solution created again a rough surface on ZnSe. In none of these cases could a reaction be controlled or a meaningful study conducted. Figure 3 shows the etching depth of ZnCdSe with varying concentrations of bromine in methanol. In all experiments, the concentration of bromine was 20 to 30 times higher than that used for ZnSe. We observed that even with these higher bromine concentrations, the etching depth was much lower than what would normally be expected for ZnSe. A log-log plot of the rate versus bromine gives a slope closer to unity, indicating roughly a first-order process up to a certain concentration of the solution. Beyond that concentration, the plot for ZnCdSe shown in

Figure 2. Etchant concentration of bromine-methanol vs etching depth rate for ZnSe grown on InP. Legend indicates total etching time.

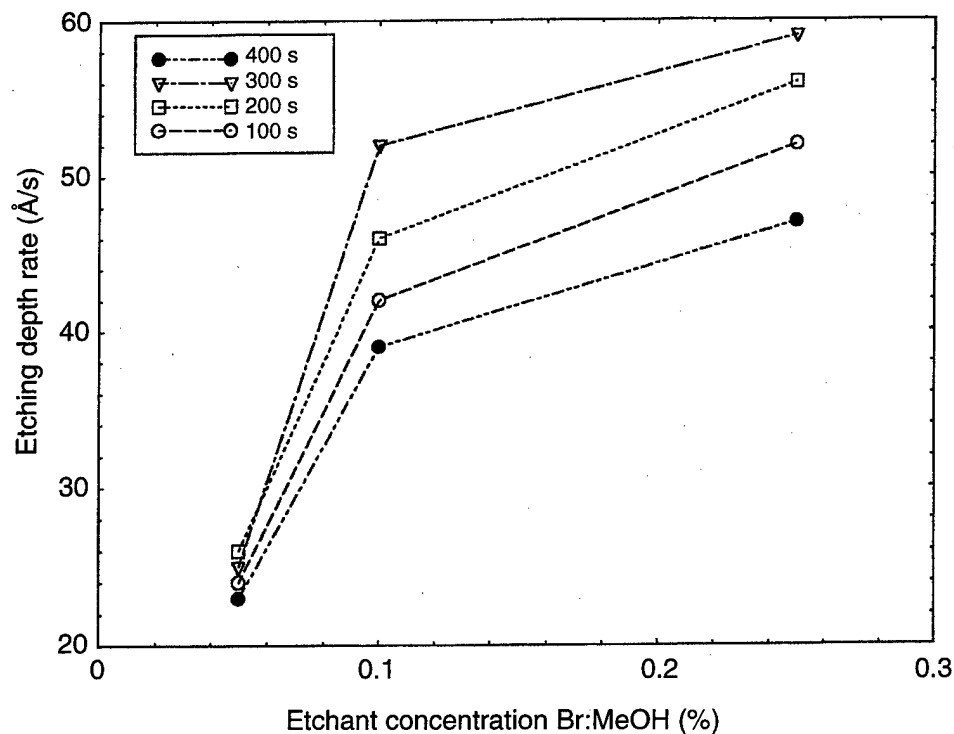


Figure 3. Etching depth as a function of bromine-methanol concentration for ZnCdSe grown on InP.

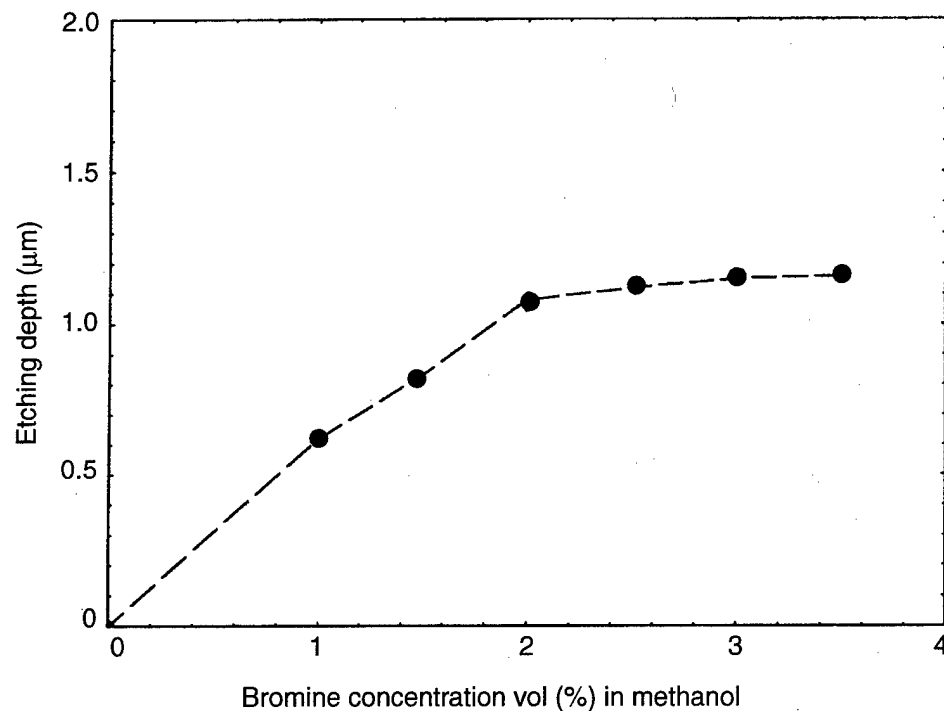


figure 3 tends to level off. This can be partially attributed to the presence of Cd in the epilayer. However, for ZnCdMgSe, a similar process could not be observed.

In general, we found that even with higher concentrations of bromine-methanol solution, ZnCdSe had little surface damage. In the same way,

the etching had no appreciable effect on ZnCdMgSe even with higher concentration and longer time of exposure. Bromine-methanol was the highest of all etchants for the epi structures, especially for ZnSe, indicating moderate solubility of Se in the solution. Table 2 lists various reagents used for ZnCdSe and ZnCdMgSe.

Sample pieces of ZnCdSe and ZnCdMgSe were dipped in the Hahnert-Schenk solution, for varying times. This solution generally distinguishes different types of dislocations. A 5-, 10-, and 15-s dipping of either ZnCdSe or ZnCdMgSe did not show any triangular or circular etch pits. Generally, ZnCdSe showed a rough surface and a few pits without any control, such that no consistency existed and results could not be reproduced. Figure 4 shows an image of ZnCdSe sample treated with Hahnert-Schenk solution.

We used a Nakagawa solution (a standard etchant for CdTe and CdZnTe (111)A) for the etching of ZnCdSe and ZnCdMgSe. Samples were treated for 1 min with vigorous agitation. We observed a few randomly distributed pits. Even with increased immersion time, the number of pits did not increase. In both cases, it was impossible to produce meaningful etch pits, and surfaces were strongly dissolved. We further treated the samples with 30% boiling NaOH. The surface reaction was quick with a few crater-like structures and with erosion and cracking appearing on ZnCdSe when we treated the samples for 1 min or more. Generally, boiling NaOH (30 wt %) leaves a high density of pits on the epitaxial surface of ZnSe, as observed by Park et al [9], but for ZnCdSe and

Table 2. Surface morphology for ZnCdSe and ZnCdMgSe grown on InP (100) after chemical etching under varying conditions of time, concentration, and temperature.

Etchant	Chemical etch	Action of etchants
Br:MeOH	1.0%, 1.5%, 2.0%, 2.5%, 3.0%, and 3.5%	Reasonable etching on ZnCdSe but little effect on ZnCdMgSe.
Hahnert-Schenk solution ^a	1:1:1 HF:chromic acid:HCl	Few pits formed on ZnCdSe. Generally rough surface. No control.
Nakagawa solution ^b	3:2:2 HF:H ₂ O ₂ :H ₂ O	Few pits formed on ZnCdSe.
Boiling NaOH (30 wt %)	—	Pits formed on ZnCdSe. No control.
HCl	—	Generally smooth surface on both ZnCdSe and ZnCdMgSe.
Polisar solution ^c	1:1:1 HNO ₃ :HCl:H ₂ O and 0.9 mL Br ₂	Smooth surface on both ZnCdSe and ZnCdMgSe.

^aI. Hahnert and M. Schenk, *J. Cryst. Growth* **101** (1990), 251.

^bK. Nakagawa, K. Maeda, and S. Takeuchi, *Appl. Phys. Lett.* **34** (1979), 574.

^cE. L. Polisar, E. M. Boinikh, G. V. Indenbaum, A. V. Vanyukov, and V. P. Schastlivii, *Izv. Vyssh. Uchebn. Zaved. Fiz.* **6** (1968), 81.

ZnCdMgSe, this was not so. Figure 5 shows the SEM image of ZnCdSe treated with boiling NaOH for 1 min.

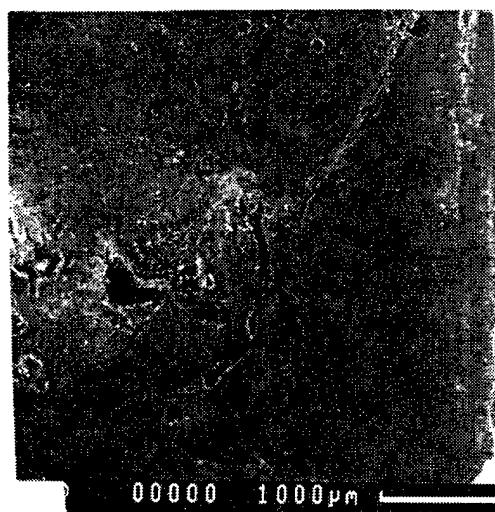
We also treated samples in HCl for different periods of time. For ZnCdSe, a few crater-like structures and erosion appeared when the samples were treated for 3 min in HCl. The surface had no major damage but was colored a light pink and yellow that quickly disappeared. A 5-min dipping in HCl created more of the same for ZnCdSe and ZnCdMgSe. A substantial bumpy pattern was developed on ZnSe with (111)A and (111)B orientations. Figure 6 shows the SEM image of ZnCdSe after 3 min of etching in HCl.

However, in the case of ZnCdMgSe, reactions were minimal when any of the etchant was used under similar conditions. Before we performed the etching studies, these samples were used for double crystal x-ray rocking curve measurements for which we used a $400\text{ CuK}\alpha_1$ reflection of radiation to find the FWHM values, which generally show how good the samples are and also give the density of lattice defects. We found that

Figure 4. SEM image of ZnCdSe treated with Hahnert-Schenk solution for 10 s.



Figure 5. SEM image of ZnCdSe treated with boiling NaOH (30 wt %) for 1 min.



samples with low FWHM also had the lowest EPD. Dislocation density (\bar{D}) was calculated using the FWHM of the crystal (F) in radians, FWHM of the monochromator crystal in radians, and the Burgers vector \mathbf{B} [11]:

$$D = (F^2 - f^2) / 9 \mathbf{B}^2. \quad (1)$$

By using the Burgers vector for a zincblende-type structure as

$$\mathbf{B} = a/2^{1/2}, \quad (2)$$

we found that as the FWHM increased, the calculated dislocation density and the experimental EPD for ZnSe also increased, as seen in figure 7.

Figure 6. SEM image of ZnCdSe after 3 min of etching with HCl.

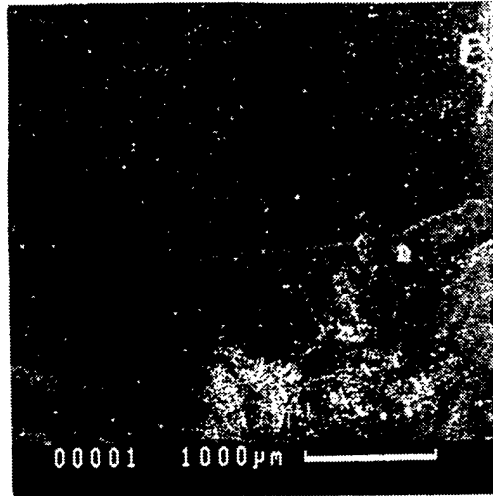
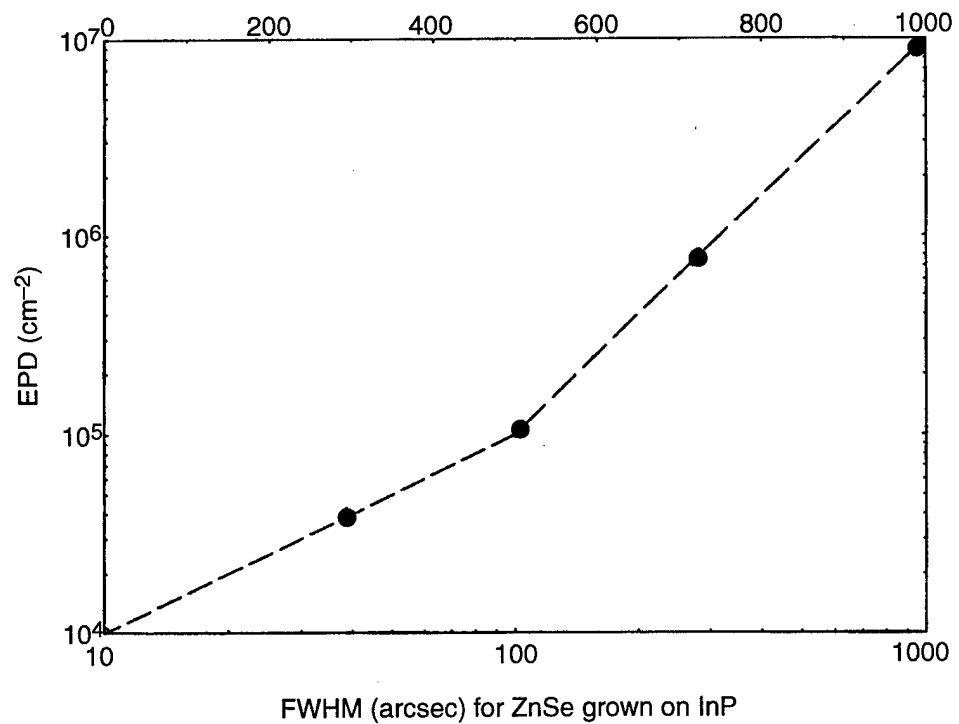


Figure 7. FWHM of x-ray rocking curves and EPD of ZnSe grown on InP (100).



4. Conclusions

The effect of chemicals under different conditions of time, concentration, and temperature on the surface of ZnSe, ZnCdSe, and ZnCdMgSe grown lattice-matched on InP has been studied. These materials exhibited good PL properties and good crystalline quality. A bromine-methanol solution with various concentrations had a reasonable etching effect on ZnSe. We studied in detail the etching rate of ZnSe for varying concentrations and etching time, and we found that the etching behavior could be controlled with a bromine-methanol solution. However, bromine-methanol had only a marginal effect on ZnCdSe and negligible effect on ZnCdMgSe. In general, the bromine-methanol solution only had a polishing effect on these samples.

When the Hahnert-Schenk solution was used on these binary, ternary, and quaternary samples, the samples showed a few circular etch pits, but the acid with low H_2O content nonuniformly dissolved the surface of these materials. The Nakagawa solution had no appreciable effect on ZnCdSe or ZnCdMgSe other than generating a few random pits and causing a strong surface dissolution. Treating these samples with boiling NaOH (30 wt %) created crater-like structure, erosion, and cracking on the surface of ZnCdSe but not on ZnCdMgSe. We observed no large number of pits. Treating the samples in HCl generally produced a bumpy pattern in ZnSe. However, nothing similar was observed with ZnCdSe and ZnCdMgSe, except some discoloration and minor erosion. Etch pit studies can be done on ZnSe with a low-concentration bromine-methanol solution with reasonable control for pit formation. For ZnCdSe and ZnCdMgSe, no meaningful pits formed with the bromine-methanol, Hahnert-Schenk, Nakagawa, or boiling NaOH (30 wt %) solutions, or HCl except surface damage, random pits, erosion, and cracking.

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